

**Loomasööt. Elavhõbeda määramine  
külmauruaatomabsorptsioonspektromeetriga (KD-AAS)  
pärast mikrolainete surve digereerimist (ekstraheerimist  
65 % lämmastikhappega ja 30 % vesinikperoksiidiga)**

**Animal feeding stuffs - Determination of mercury by  
cold-vapour atomic absorption spectrometry (CVAAS)  
after microwave pressure digestion (extraction with 65  
% nitric acid and 30 % hydrogen peroxide)**

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

See Eesti standard EVS-EN 16277:2012 sisaldab Euroopa standardi EN 16277:2012 ingliskeelset teksti.	This Estonian standard EVS-EN 16277:2012 consists of the English text of the European standard EN 16277:2012.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 04.07.2012.	Date of Availability of the European standard is 04.07.2012.
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ICS 65.120

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ICS 65.120

English Version

**Animal feeding stuffs - Determination of mercury by cold-vapour  
atomic absorption spectrometry (CVAAS) after microwave  
pressure digestion (extraction with 65 % nitric acid and 30 %  
hydrogen peroxide)**

Aliments des animaux - Dosage du mercure par  
spectrométrie d'absorption atomique à vapeur froide  
(SAVVF) après digestion sous pression par micro-ondes  
(extraction avec de l'acide nitrique à 65 % et du peroxyde  
d'hydrogène à 30 %)

Futtermittel - Bestimmung von Quecksilber mit Kaltdampf-  
Atomabsorptionsspektrometrie (KD-AAS) nach  
Mikrowellen-Druckaufschluss (Extraktion mit 65 %  
Salpetersäure und 30 % Wasserstoffperoxid)

This European Standard was approved by CEN on 17 May 2012.

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## Foreword

This document (EN 16277:2012) has been prepared by Technical Committee CEN/TC 327 “Animal feeding stuffs – Methods of sampling and analysis”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2013, and conflicting national standards shall be withdrawn at the latest by January 2013.

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## 1 Scope

This European Standard specifies a method for the determination of mercury in animal feeding stuffs by Cold-Vapour Atomic Absorption Spectrometry (CVAAS) after microwave pressure digestion. The limit of quantification in the test solution should be 0,25 µg/l or lower. Using a test portion of 0,5 g and a volume of the test solution of 25 ml a limit of quantification of 0,0125 mg/kg or lower should be obtained.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

EN ISO 6497, *Animal feeding stuffs — Sampling (ISO 6497)*

EN ISO 6498, *Animal feeding stuffs — Guidelines for sample preparation (ISO/FDIS 6498)*

## 3 Principle

Mercury is determined in the test solution by cold-vapour atomic absorption spectrometry (CVAAS) after microwave pressure digestion.

The homogenised feeding stuff test sample is digested with nitric acid and hydrogen peroxide under pressure and high temperatures in a microwave-heated pressure digestion system.

The test solution is transferred to the reaction vessel of the mercury analysis unit. The mercury is reduced with sodium borohydride or tin(II) chloride to elemental volatile mercury and flushed into the cell of the AAS instrument using a carrier gas stream. As an option with an additional amalgamation step, sensitivity could be increased and matrix effects could be decreased. The absorption at 253,7 nm (mercury line) is used as a measure of the mercury concentration in the cell.

Other digestion procedures with the same extraction efficiency (see Annex D and Annex E) or other measurement systems like FI-CVAAS (flow injection cold-vapour atomic absorption spectroscopy) or CV-ICP-AES (cold-vapour inductively coupled plasma atomic emission spectroscopy) are possible.

**WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.**

## 4 Reagents

The concentration of the trace elements in the reagents and water used shall be low enough not to affect the results of the determination. A blank should be measured simultaneously with the test samples on each day of analysis to control contamination and carry over with mercury in the reagents and apparatus used.

Use water conforming to grade 2 of EN ISO 3696.

**4.1 Nitric acid (HNO<sub>3</sub>),** not less than 65 % (mass fraction), of approximately  $\rho(\text{HNO}_3) = 1,4$  g/ml.

NOTE Use nitric acid available with high purity or perform a sub-boiling distillation.

**4.2 Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>),** not less than 30 % (mass fraction).